STEREOCHEMICAL STUDIES. XV*

t-Butylcyclopentane Derivatives. II**
Synthesis of *cis*- and *trans*-2-*t*-butylcyclopentanol and *cis*- and *trans*-3-*t*-butylcyclopentanol

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(Received July 4, 1972)

2-*t*-Butylcyclopentanone (**6**) was prepared by anomalous Grignard reaction of 2-*iso*-propylidenecyclopentanone (**5**) with CH_3MgI. Reduction of **6** with lithium aluminium hydride gave 54% *cis*- and 46% *trans*-2-*t*-butylcyclopentanol (**1**, **2**). The isomers were separated by vpc.

Stereospecific synthesis of *cis*- and *trans*-3-*t*-butylcyclopentanol (**3**, **4**) has been performed from *cis*- and *trans*-4-*t*-butylcyclopentene-1,2-oxide (**8**, **9**) by reduction with lithium aluminium hydride. Synthesis of several other *t*-butylcyclopentane derivatives is also described.

While *t*-butylcyclohexane derivatives were intensively investigated and furnished interesting data concerning the relations between steric structure, conformation and reactivity [1—7], only some publications [8—10] deal with the synthesis and reactivity of analogous *t*-butylcyclopentane derivatives. In an earlier paper we described [11] the stereospecific synthesis of the four 2-amino-4-*t*-butylcyclopentanol isomers. The present work is concerned with the synthesis of *cis*- and *trans*-2-*t*-butylcyclopentanol (**1**, **2**) as well as of *cis*- and *trans*-3-*t*-butylcyclopentanol (**3**, **4**), discussing also the synthesis of several further *t*-butylcyclopentane derivatives.

As a general method for the synthesis of 2-alkyl substituted cyclopentanones, cleavage of reaction products of 2-carbethoxycyclopentanone enol potassium salts with alkyl halogenides can be used [12]. This is a convenient method for the preparation of *n*-alkyl substituted cyclopentanones.

An interesting method of preparing 2-*t*-alkylated cyclopentanones and cyclohexanones was recently described by HENNION and QUINN [13]. Alkylation of the pyrrolidine enamines derived from cyclopentanone and cyclohexanone with *t*-propargylic chlorides produced, after hydrolysis, the corresponding 2-*t*-alkylated cyclanones. Using this method, the preparation of 2-*t*-amylcyclopentanone is described, however, the preparation of 2-*t*-butylcyclopentanone is not mentioned.

* Part XIV.: P. Sohár, G. Bernáth: Org. Magnetic Resonance, in press.

** Part I.: G. Bernáth, M. Svoboda: Tetrahedron **28**, 3475 (1972).

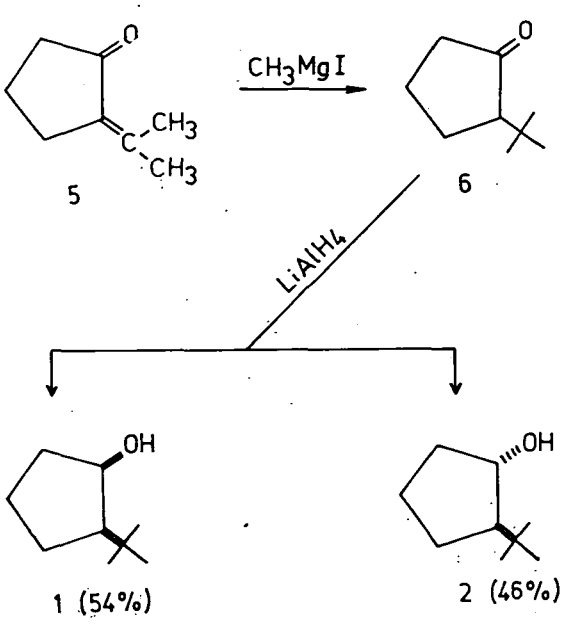

Fig. 1

Reacting 2-isopropylidenecyclopentanone (**5**) [14] with methyl magnesium iodide in the presence of CuCl, an anomalous Grignard reaction [15] takes place, giving 2-*t*-butylcyclopentanone (**6**) with a good yield (Fig. 1). The reduction of 2-*t*-butylcyclopentanone was performed with lithium aluminium hydride and yielded 54% *cis*- and 46% *trans*-2-*t*-butylcyclopentanol (**1**, **2**). This isomer ratio fits well to the value calculated by the Ugi—Ruch equation [16] for the reduction of a series of 2-substituted cyclopentanone derivatives with lithium aluminium hydride [17] and is in accordance with the recent results of HENNION and QUINN [13], who found in the reduction of 2-substituted cyclopentanones with $NaBH_4$ that the ketones bearing the bulkliest groups produce the *cis* isomer preferentially, whereas those bearing relatively small groups give the *trans* isomer as main product.

The isomeric alcohols (**1**, **2**) were separated by vpc.

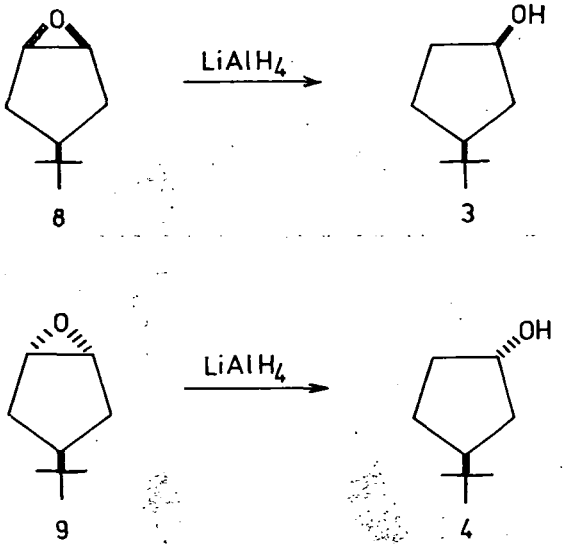

Fig. 2

Cis- and *trans*-3-*t*-butylcyclopentanol (**3**, **4**) can be obtained from *cis*- and *trans*-4-*t*-butylcyclopentene-1,2-oxide (**8**, **9**) with lithium aluminium hydride (Fig. 2). We suceeded in the stereospecific synthesis of both isomeric epoxide starting from 4-*t*-butylcyclopent-1-ene (**7**) [11].

RICHTER and GILARDEAU [9] separated the *cis*-3-*t*-butylcyclopentanol (**3**) from the *cis-trans* mixture obtained with catalytic reduction of 3-*t*-butylcyclopentanone by fractionated crystallization of the 3,5-dinitrobenzoates. By dehydration of *cis*- and *trans*-3-*t*-butylcyclopentanol they obtained 3- and 4-*t*-butylcyclopent-1-ene and succeeded in separating the 4-*t*-butylcyclopent-1-

ene from this mixture by preparative gas chromatography. The epoxide mixture obtained from this olefine was reduced with lithium aluminium hydride and the *trans* isomer separated from the *cis-trans* alcohol mixture by combining preparative gas chromatography and fractional crystallization of the *p*-nitrobenzoates. The configurations were determined by comparing the IR spectra of the 3-*t*-butylcyclopentanols obtained with those of 3-methylcyclopentanol and 3-*iso*-propylcyclopentanol.

For the preparation of 4-*t*-butylcyclopent-1-ene, we found the synthesis *via* 2-carbethoxy-4-*t*-butylcyclopentanone (**10**) → 2-carbethoxy-4-*t*-butylcyclopentanol → 2-hydroxy-4-*t*-butylcyclopentanecarboxylic acid → 4-*t*-butylcyclopent-1-ene (**7**) as the most convenient. In the present paper an alternative method of preparing **7**, using also 2-carbethoxy-4-*t*-butylcyclopentanone (**10**) as starting material, and based on the fact, that 1,2-dicarboxylic acids can be decarboxylized to olefine by lead tetraacetate [18] is discussed. Therefore, the preparation of 4-*t*-butylcyclopentene-1,2-dicarboxylic acid (**19**) was attempted from 2-carbethoxy-4-*t*-butylcyclopentanone (**10**) on the analogy of the synthesis of cycloheptane-1,2-dicarboxylic acid [19] (Fig. 3).

Hydrogen cyanide addition to 2-carbethoxy-4-*t*-butylcyclopentanone (**10**) was effected with liquid hydrocyanic acid at 0 °C. The dehydration of the HCN-adduct (**11**) was accomplished in pyridine with phosphorus oxychloride. The 1-cyano-2-carbethoxy-4-*t*-butylcyclopentene obtained was found to be homogeneous by gas chromatography. The reaction product was supposed to consist only of the isomer (**12**) containing the double bound in 1,2 position, as the presence of 2-cyano-3-carbethoxy-5-*t*-butylcyclopent-1-ene (**13**) could not be detected. However, after esterification of the olefin-dicarboxylic acid obtained from the nitrile by 40-hour hydrolysis with 40% potassium hydroxide, gas chromatographic analysis revealed the presence of the ethyl esters of two acids (**14, 15**) in a ratio 56%:46%. The mixture of the olefinic acids was converted into the anhydrides (**16, 17**) by treatment with acetic anhydride. After conversion of the anhydride mixture into the ethyl ester, the gas chromatogram showed only one peak, supposedly corresponding to the diethyl ester of (**14**).

Hydrogenation of the unsaturated anhydride (**16**) unequivocally gives the 4-*t*-butylcyclopentane-1,2-dicarboxylic anhydride (**18**), from which **7** can be obtained on the analogy of the methods described previously [15]. Though it seemed more convenient for the production of 4-*t*-butylcyclopent-1-ene to apply the method described above using the decarboxylation of 4-*t*-butylcyclopent-1-ene-1-carboxylic acid, it seemed worth while to mention this way too, because the unsaturated anhydride (**16**) described and the saturated compound (**18**) which may be obtained by hydrogenation are potentially useful starting materials for the synthesis of numerous 1,2-disubstituted-4-*t*-butylcyclopentane derivatives.

The stereospecific synthesis of the *cis* epoxide **8** was achieved by alkaline treatment of the acetyl hypobromite adduct of **7**. The stereohomogeneous *trans* compound (**9**) could be obtained by quaternization of 2-*trans*-dimethylamino-*trans*-4-*t*-butylcyclopentanol with methyl iodide and liberation of the base followed by treatment at 150 °C. The IR spectra of the *cis*- and *trans*-epoxides (**8, 9**) showed marked differences in the region 800—1400 cm^{-1}, while at higher wave numbers they are very similar.

The *cis*- and *trans*-3-*t*-butylcyclopentanols (**3, 4**) could be prepared from the

stereohomogeneous *cis*- and *trans*-epoxides (**8**, **9**) by lithium aluminium hydride reduction with very good yield.

As another way for the stereospecific synthesis of *cis*-3-*t*-butylcyclopentanol (**3**), the transformation of *cis*-2-dimethylamino-*cis*-4-*t*-butylcyclopentanol (**21**) into the N-oxide **22**, and hydrogenation of the 3-hydroxy-*cis*-5-*t*-butylcyclopent-1-ene (**23**) obtained by thermolysis of the N-oxide seemed also suitable (Fig. 4). Though the IR spectrum of the non-basic portion of the reaction product was consistent, with the structure of 3-hydroxy-*cis*-5-*t*-butylcyclopent-1-ene, without any sign of the presence of saturated ketone, this method proved unsatisfactory for a practical synthesis of *cis*-3-*t*-butylcyclopentanol, in consequence of the poor yield of the pyrolysis (giving *cis*-2-dimethylamino-*cis*-4-*t*-butylcyclopentanol (**21**) as the main product). Nevertheless, considering the rule of *cis* elimination, the above fact furnished a supporting piece of evidence for the steric structure of *cis*-2-amino-*cis*-4-*t*-butylcyclopentanol (**20**) proved also by other independent ways [11].

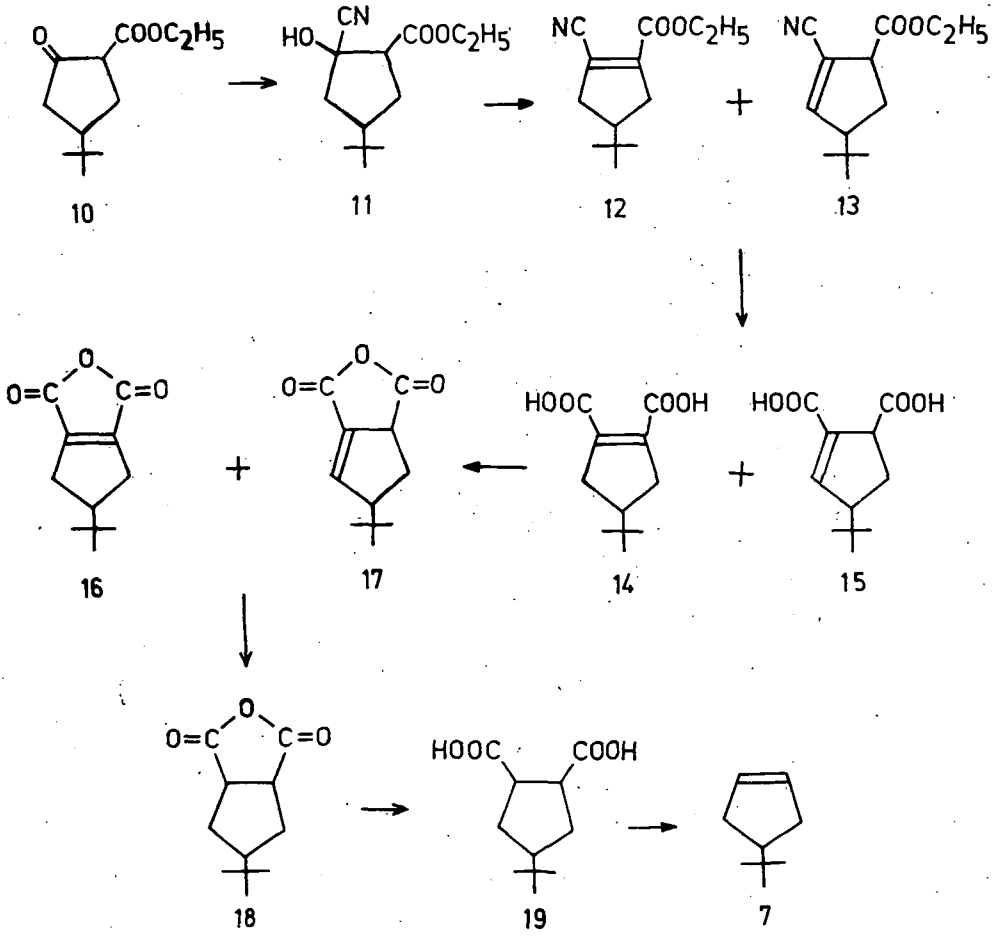

Fig. 3

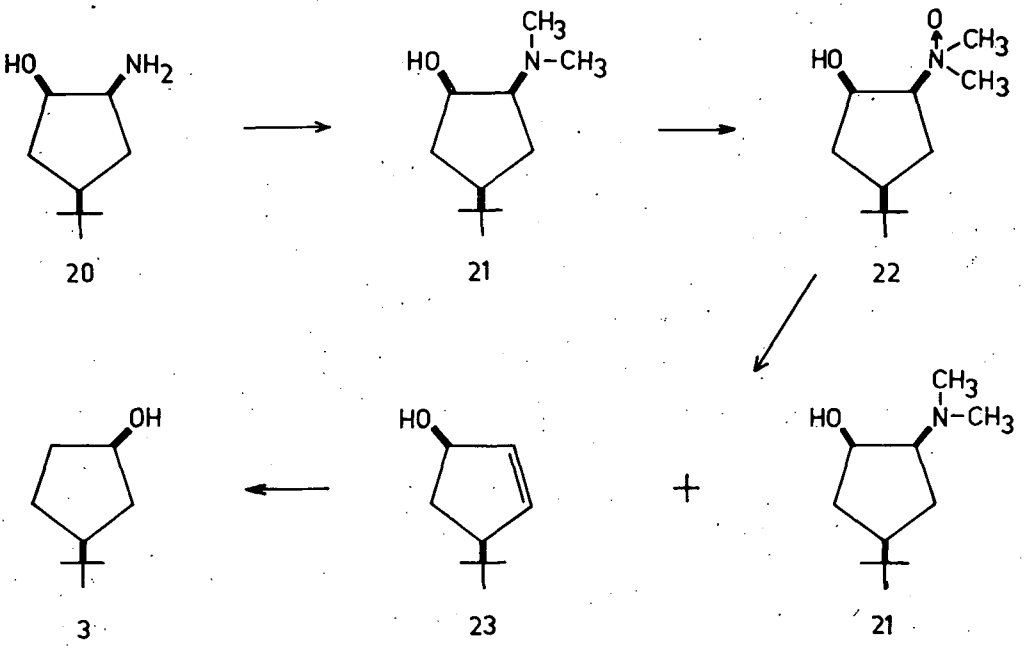

Fig. 4

Experimental

2-t-*Butylcyclopentanone* (**6**)

2-*iso*-Propylidenecyclopentanone (**5**) was prepared from aceton and cyclopentanone in 1 *N* sodium hydroxyde solution, according to the procedure of CONIA and SANDRE [14]. The crude product was carefully fractionated and the main crop collected at 76—78 °C (11 torr).

A Grignard reagent was prepared from 4.8 g of magnesium turnings and 32.0 g of methyl iodide in 50 ml of dry ether. When the reaction proceeded the mixture was cooled and 0.3 g of freshly prepared CuCl was introduced. To this solution 18.6 g of 2-*iso*-propylidenecyclopentanone in 50 ml of dry ether was added at a rate to maintain the temperature between −5—0 °C. After removal of the cooling bath, stirring was continued for 30 minutes. The reaction mixture was poured into 250 ml of ice water containing 6 ml of conc. sulfuric acid. The ethereal layer was separated, the aqueous layer extracted with 3×50 ml of ether. The combined ethereal solution was washed with 2×50 ml of water, dried on magnesium sulfate. On evaporating the ether, 10.8 g of 2-*t*-butylcyclopentanone (**6**) (54%) was obtained. The 2,4-dinitrophenylhydrazone melted at 156—158 °C. The product was found to be contaminated with small amounts of unidentified materials.

$C_{15}H_{20}N_4O_4$ (320.36). Calcd. C 56.24; H 6.29. Found C 56.05; H 6.34%.

Cis- *and* trans-2-t-*Butylcyclopentanol* (**1**, **2**)

2.8 g of 2-*t*-butylcyclopentanone (**6**) purified by vpc was treated with excess lithium aluminium hydride (0.56 g in 25 ml of ether). The hydrolyzed reaction mixture was worked up in the usual manner and the isomeric alcohols were separated by vpc, using a 1 cm×6 cm column packed with 20% diglycerol on Chromosorb W at 90 °C.

IR spectrum: *cis*-2-*t*-butylcyclopentanol νOH 3435 cm^{-1}, νC—O 1012 cm^{-1}; *trans*-2-*t*-butylcyclopentanol νOH 3350 cm^{-1}, νC—O 1026 cm^{-1}. NMR spectrum: *cis*-2-*t*-butylcyclopentanol C—1 H: $\delta = 4.1$ ppm; *trans*-2-*t*-butylcyclopentanol C—1 H: $\delta = 3.9$ ppm.

The reduction mixture consisted of 54% *cis*- and 46% *trans*-2-*t*-butylcyclopentanol (**1**, **2**). The alcohols were converted to 3,5-dinitrobenzoate.

Cis-3,5-dinitrobenzoate m.p. 131—133 °C. $C_{16}H_{20}N_2O_6$ (336.36). Calcd. C 57.14; H 5.99. Found C 56.78; H 5.72%.

Trans-3,5-dinitrobenzoate m.p. 89—91 °C. $C_{16}H_{20}N_2O_3$ (336.36). Calcd. C 57.14 H 5.99. Found C 56.87; H 5.71%.

1-*Cyano*-2-*carbethoxy*-4-t-*butylcyclopentanol* (**11**)

80 ml of ethanol was cooled to 0 °C, and 1 ml of a saturated aqueous potassium cyanide solution, then 50 g of hydrocyanic acid cooled to 0 °C were added. To this cyanide solution, maintained at 0 °C, 30.3 g (0.143 mole) of 2-carbethoxy-4-*t*-butylcyclopentanone (**10**) dissolved in 50 ml ethanol was added dropwise. The reaction mixture was allowed to stand overnight at +4 °C, then at room temperature for 24 hrs. After addition of 200 ml of ethanol, the reaction mixture was neutralized with aqueous oxalic acid solution, filtered, and the ethanol and the excess of hydrogen cyanide were evaporated. The residue was taken up in 400 ml of ether, dried over anhydrous sodium sulfate, filtered, and the ether evaporated to leave 33.1 g (96.9%) of a brown oil. Distilling a small portion at 30 torr, a thick oil ($n_D^{20} = 1.4553$) was obtained. The nitrogen content of the product was somewhat lower than calculated for **11**.

$C_{13}H_{21}O_3N$ (239.31). Calcd. N 5.85. Found N 5.33%. This crude reaction product of the hydrogen cyanide addition was used in the following experiments.

1-*Cyano*-2-*carbethoxy*-4-t-*butylcyclopent*-1-*ene* (**12**)

To 31.6 g of 1-cyano-2-carbethoxy-4-*t*-butyl-cyclopentanol (**11**) resulting from the above reaction, 100 ml of abs. pyridine and 100 ml of abs. benzene were added. The mixture was cooled to +3 °C and 60 ml phosphorus oxychloride dissolved in 80 ml abs. pyridine was added, maintaning the temperature between 3 and 10 °C. Separation of some solid by-product was observed. The reaction mixture was allowed to stand at room temperature overnight, then heated to boiling and kept at this temperature for 10 min. After cooling to room temperature, the reaction mixture was poured onto ice, then extracted with benzene. The extract was washed with 10% H_2SO_4, with 10% $NaHCO_3$ solution, finally twice with water, dried over anhydrous magnesium sulfate, and the benzene distilled off. 24.3 g (82.6%) of an oil remained, which was distilled at 18 torr yieldeding 1.8 g forerun, then 18.1 g of the main fraction boiling at 202—204 °C. Gas chromatographic analysis showed

the presence of only one product, supposedly 1-cyano-2-carbethoxy-4-*t*-butylcyclopent-1-ene (**12**). The presence of 2-cyano-3-carbethoxy-5-*t*-butylcyclopent-1-ene (**11**), the formation of which is possible in principle, could not be detected. The product was transformed without further purification into 4-*t*-butylcyclopent-1-ene-1,2-dicarboxylic acid.

4-t-*Butylcyclopent*-1-*ene*-1,2-*dicarboxylic acid* (**14**) *and* 5-t-*butylcyclopent*-1-*ene*-2,3-*dicarboxylic acid* (**15**)

14.7 g of (**12**) was refluxed with 100 ml of 40% potassium hydroxyde solution and 30 ml of ethanol for 5 hrs., then the ethanol was evaporated and the reaction mixture refluxed for further 8 hrs. To remove the impurities, the cooled solution was extracted with ether, acidified with conc. HCl and extracted with ether. The ethereal solution was washed with water, dried over anhydrous magnesium sulfate and the ether evaporated. 12.1 g (87.8%) of a product was obtained, which, on recrystallization from ether—petroleum ether, gave crystals melting over a rather wide range at 110—115 °C. Gas chromatography of the product obtained from this material by esterification with diazomethane, showed the presence of two main products (34.8%:65.2% in the order of rising retention times), which were supposed to be methyl esters of the carboxylic acids **14** and **15**. As the isomers could not be separated by recrystallization, and hydrogenation eliminating the difference, which is therefore irrelevant in the further synthesis, gave the same dicarboxylic acid, the product was subjected to acetic anhydride treatment to prepare the anhydride.

4-t-*Butylcyclopent*-1-*ene*-1,2-*dicarboxylic anhydride* (**16**)

10.0 g of the above rough dicarboxylic acid (**14**, **15**) was refluxed with 200 ml of acetic anhydride for 3 hrs. The excess of acetic anhydride was removed from the reaction mixture by distillation at 30 torr. The residue was distilled at 18 torr and the fraction of b.p. 175—190 °C, consisting of a pale-yellow oil (8.3 g; 90.7%), was collected.

$C_{11}H_{14}O_3$ (194.22). Calcd. C 68.02; H: 7.27. Found C 67.80; H 7.61%.

Cis-3-t-*butylcyclopentanol* (**3**)

In a three-necked 100 ml round-bottomed flask provided with stirrer, reflux condenser and dropping funnel, 18 ml of an abs. ethereal lithium aluminium hydride solution, containing 32.7 mg $LiAlH_4$ pro ml was placed. To this solution (0.016 mole $LiAlH_4$), 1.3 g (0,0093 mole) of *cis*-4-*t*-butylcyclopentene-1,2-oxide (**8**) dissolved in 5 ml of abs. ether was added under stirring, and the mixture was refluxed for 2 hrs. The complex was then decomposed by succesive addition of 1 ml of water, 3 ml of 15% sodium hydroxide solution, and 3 ml of water, under cooling. The precipitate was filtered off, washed with ether, and the combined ethereal solutions were evaporated after drying. 1.25 g (94.8%) of a colourless liquid remained, which distilled at 30 torr at 115—117 °C to yield 1.15 g (87.2%) of **1**.

$C_9H_{16}O$ (142.23). Calcd. C 76.00; H 12.75. Found C 76.21; H 12.68%.

Trans-3-t-*butylcyclopentanol* (**4**)

From 250 mg of stereohomogeneous *trans*-4-*t*-butylcyclopentene-1,2-oxide (**9**), prepared from *trans*-2-dimethylamino-*trans*-4-*t*-butylcyclopentanol methiodide by the process described earlier [11], 220 mg (86.2%) of *trans*-3-*t*-butylcyclopentanol (**4**) was obtained.

$C_9H_{16}O$ (142.25). Calcd. C 76.00; H 12.75. Found C 76.06; H 12.85%.

Cis-2-*dimethylamino*-cis-4-t-*butylcyclopentanol* (**21**)

2.1 g of *cis*-2-amino-*cis*-4-*t*-butylcyclopentanol (**20**) was refluxed with 70 ml of formic acid and 70 ml of 36% aqueous formaldehyde for 24 hrs. The reaction mixture was evaporated to dryness under reduced pressure (30 torr), conc. aqueous potassium hydroxide solution was added and the mixture was repeatedly extracted with ether. The ethereal extract was shaken twice with 1 *N* hydrochloric acid and the acidic extract made alkaline with potassium hydroxide. The dimethylamino derivative (**21**) which separated was taken up in ether and dried over anhydrous magnesium sulfate. After evaporation of the ether, the remaining 2.1 g (84.9%) of light-yellow *cis*-2-dimethylamino-*cis*-4-*t*-butylcyclopentanol was dissolved in about 10 ml of petr. ether (b.p. 45—60 °C). Storage at −70 °C yielded 1.63 g of a white crystalline substance m.p. 60—61 °C. A small amount was twice recrystallized to give m.p. 63 °C.

The hydrochloride prepared in the usual way was recrystallized three times from abs. ethanol-ether; m.p. 228—228.5 °C.

$C_{11}H_{24}NOCl$ (221.77). Calcd. C 59.57; H 10.91. Found C 59.21; H 10.72%.

3-*Hydroxy*-5-t-*butylcyclopent*-1-*ene* (**23**)

560 mg (0.003 mole) of *cis*-2-dimethylamino-*cis*-4-*t*-butylcyclopentanol (**21**) was dissolved in 5 ml methanol, cooled to −5 °C, 5 ml of 30% H_2O_2 added and, after slowly reaching the room temperature, shaken for 5 hrs. The excess of H_2O_2 was decomposed with Pt black under nitrogen and, after filtration, the reaction mixture was evaporated at 20 torr. 600 mg (98.7%) of white crystals (**22**) remained. M.p. unsharp. A small part begins to melt at 60 °C, showing the presence of the starting amine, the main crop melts between 120—132 °C, under decomposition.

The rough product, decomposing at room temperature, was dissolved in abs. ethanol without further purification, placed in a Hickman collar flask and distilled at 10 torr. Distillation began at 170 °C bath temperature, yielding 440 mg oil up to 210 °C. The product was taken up in ether and washed with dil. HCl. The ethereal part, dried and evaporated, yielded 150 mg slightly coloured oil, which, distilled at 20 torr, gave 128 mg of the colourless reaction product (**23**) (26.1% of the starting amine), which proved to be homogeneous by gas chromatography. IR: no signs of saturated ketone.

From the HCl solution, after alkalization and etheric extraction, 270 mg (45.0%) of the starting *cis*-2-dimethylamino-*cis*-4-*t*-butylcyclopentanol (**21**) showing no m.p. depression with an authentic sample could be recovered.

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СТЕРЕОХИМИЧЕСКИЕ ИССЛЕДОВАНИЯ XV. ПРОИЗВОДНЫЕ *t*-БУТИЛЦИКЛОПЕНТАНА II. СИНТЕЗ *ЦИС*- И *ТРАНС*-2-*t*-БУТИЛЦИКЛОПЕНТАНОЛА И *ЦИС*- И *ТРАНС*-3-*t*-БУТИЛЦИКЛОПЕНТАНОЛА

Г. Бернат, Л. Грубер и И. Тёмёшкёзи

Из 2-изопропилиденциклопентанола с CH_3MgI аномальной реакцией Гриньяра получен 2-*t*-бутилциклопентанон (**6**). При восстановлении 2-*t*-бутилциклопентанона литийалюмогидридом образовалось 54% *цис*- и 46% *транс*-2-*t*-бутилпентанола (**1, 2**). Изомеры были разделены при помощи препаративной газовой хроматографии.

Стереоспецифический синтез *цис*- и *транс*-3-*t*-бутилциклопентанола (**3, 4**) был осуществлён реакцией восстановления *цис*- и *транс*-4-*t*-бутилциклопентен-1,2-оксида (**8, 9**) литийалюмогидридом. Описаны синтезы ещё некоторых производных *t*-бутилциклопентана.